

REMARKS

Applicants wish to thank Examiner Dan Jenkins for his courteous and helpful discussion with the Applicants' representatives.

Claims 1-13 were pending. Claims 3-11 were withdrawn from under consideration in the application.

In the Office Action of March 31, 2003, claims 1, 2, 12 and 13 were rejected.

Claims 1, 2, 12 and 13 were rejected under 35 U.S.C. §102(b) as being anticipated by Shindo et al. (U.S. Patent No.: 5,667,665). Applicants respectfully traverse this rejection.

In response to the in-person interview with Examiner Jenkins on August 26, 2003, Applicants hereby submit a § 132 Declaration by one of the inventors, Masahito Uchikoshi, attesting to results of the present invention which obtained a high purity cobalt with reduced copper concentration of 0.101/mass-ppm or less . Examiner Jenkins indicated in the interview that claims 1, 2, 12 and 13 would be allowed upon receipt of a §132 Declaration attesting to the comparative experimental results conducted using both the method of the present invention and the method of prior reference, Shindo et al. (U.S. Patent No.: 5,667,665).

As evidenced by the experiment, which is enclosed with this response, the present invention obtains a high purity cobalt with far less impurities than Shindo et al. The present invention produced copper monovalent ions from adjusting the concentration of hydrochloric acid from 0.1 kmol/m³ to 3 kmol/m³. Thus, copper was separated from an aqueous solution of cobalt chloride and a high purity cobalt with reduced copper concentration of 0.101/mass-ppm or less was obtained. Shindo et al. does not teach or even suggest a high purity cobalt with reduced copper concentration of 0.101/mass-ppm.

Accordingly, Applicant submits that the claimed invention is not anticipated by the applied references as suggested by the Examiner.

In view of the foregoing, it is submitted that all remaining claims are patentable and that the application is in condition for allowance. Notice to that effect is respectfully requested.

Respectfully submitted,

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT(S): Uchikoshi et al. DOCKET NO.: 09799107-0009
SERIAL NO.: 09/966,861 GROUP ART UNIT: 1742
DATE FILED: September 28, 2001 EXAMINER: DANIEL JENKINS
TITLE: "HIGH PURITY COBALT, METHOD OF MANUFACTURING
THEREOF, AND HIGH PURITY COBALT TARGETS"

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DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Commissioner:

I declare that I am one of the inventors of pending application Serial Number 09/966,861, filed September 29, 2001.

I hereby declare as follows:

An experiment was conducted using both the method of the present invention and the method of Shindo et al. (U.S. Patent No.: 5,667,665). The results showed that the present invention obtains a high purity cobalt with far less impurities than Shindo et al. Figures and charts are attached to this declaration.

The following is a description of the experiment using the method of the present invention and the method of Shindo et al. References should be made to the attached Figures 1-9, incorporated herein by reference. The results are summarized in Table 1, also incorporated herein by reference.

A. Shindo et al's Method

First, a 600g crude cobalt lump with a general gettable purity was charged into a vessel containing about 12.5 liters of 11.6N aqueous solution of hydrochloric acid. The temperature

was raised to 95 °C, and after 7 hours an aqueous solution of cobalt chloride having a hydrochloric acid concentration of 9N and a cobalt concentration of 50 g/l was obtained.

As shown in FIG. 4, 12 liters of this solution was passed through a polypropylene column (150 mm diameter x 1200 mm length) filled up with 12 liters of anion exchange resin to allow the cobalt to absorb to the resin followed by washing with 12 liters of 9N hydrochloric acid.

Next, 18 liters of 4N hydrochloric acid was passed through the column to elute cobalt. FIG. 6 shows changes in the concentrations of metal ions in the effluent (elution curve). In FIG. 5, the abscissa represents the volume of effluent and the ordinate represents the maximum concentrations of the metal ions.

The equilibrium distribution coefficient of Fe, Co, Ni, and Cu to anion exchange resins is shown in FIG. 7.

A lot of peaks of the elution curves of the divalent cobalt ions and the divalent copper ions overlap, so copper cannot be completely separated from the aqueous solution of cobalt chloride. That is, copper exists in the recovered aqueous solution of cobalt chloride.

The recovered aqueous solution of cobalt chloride was vaporized to dryness at 140 °C using a rotary evaporation device. 1600 g of vaporized and dried material was obtained as $\text{CoCl}_2 \cdot 2\text{H}_2\text{O}$. This material was dissolved into pure water to obtain 10 liters of solution having a cobalt concentration of 60 g/l. After adjusting its pH to 1, 5 liters of the purified cobalt solution was charged into an electrolytic cell. The remainder of the solution was used as a catholyte supplying solution.

Next, electrolytic refining was conducted at a current density of 0.02 A/cm^2 and 50 °C using a crude cobalt sheet as a cathode. The anode side and the cathode side of the cell were partitioned with a diaphragm. The purified aqueous solution of cobalt chloride was supplied at the feed rate of 120 ml/hr in the cathode side while withdrawing the purified aqueous solution of

cobalt chloride from the anode side at the same flow rate as in the cathode side. After 40 hours, 83g electro-deposited cobalt was obtained with a yield of 95%. The electro-deposited cobalt was subjected to electron beam melting to obtain 79g high purity cobalt. The purity of the cobalt obtained from the above operations is shown in Table 1. Table 1 shows about 30/mass-ppm of copper as an impurity.

B. The Method of the Present Invention:

FIGS. 1 and 2 show a flow chart of a manufacturing process employing principles of the invention. First, in order to prepare an aqueous solution of cobalt chloride, generally gettable crude cobalt was dissolved into 1.5 kmol/m^3 of hydrochloric acid solution until the concentration of cobalt reached 0.34 kmol/m^3 (20 g/dm^3) (Step S101). Then, powdered cobalt was added to the aqueous solution of cobalt chloride, and an inert gas was injected into the solution with agitating, as shown in FIG. 3, to convert divalent copper ions to monovalent copper ions (Step S102). Then, as shown in FIG. 4, the aqueous solution of cobalt chloride was contacted with anion exchange resins to absorb the copper and separate the copper from the aqueous solution of cobalt chloride (Step S103). After separating the copper, the concentration of hydrochloric acid of the aqueous solution of cobalt chloride was adjusted to 9 kmol/m^3 , and the aqueous solution of cobalt chloride was contacted with the anion exchange resins to absorb the cobalt and separate impurities such as titanium (Step S104). After that, the cobalt was eluted from the column filled up with the anion exchange resins with 4 kmol/m^3 of hydrochloric acid solution to separate impurities such as molybdenum (Step S105).

FIG. 8 shows changes in the concentrations of metal ions in the effluent (elution curve). In FIG. 9, the abscissa represents the volume of effluent and the ordinate represents the maximum concentrations of the metal ions.

The equilibrium distribution coefficient of Fe, Co, Ni and Cu to anion exchange resins is shown in FIG. 7.

The peaks of the elution curves of the divalent cobalt ions and the divalent copper ions do not overlap, so copper can be completely separated from the aqueous solution of cobalt chloride. That is, no copper exists in the recovered aqueous solution of cobalt chloride.

After eluting the cobalt from the column filled up with the anion exchange resins, the obtained aqueous solution of cobalt chloride was evaporated to dryness to obtain cobalt chloride or hydrates thereof (Step S106). Then, as shown in FIG. 5, the obtained cobalt chloride or hydrates thereof were heated to 743K in a hydrogen atmosphere to obtain cobalt (Step S107). The obtained cobalt was molten with plasma arc containing active hydrogen to remove impurities such as oxygen (Step S108) to obtain high purity cobalt.

Quantities of impurities contained in the obtained high purity cobalt were determined by Glow Discharge Mass Spectroscopy and the impurity was calculated. Table 1 shows the results. As shown in Table 1, the copper concentration was as very low as 0.101 /mass-ppm or less, and the value of purity was very high.

C. Results:

As can be seen from Table 1, the present invention obtained high purity cobalt with far lesser impurities than the Shindo et al. method. The method of Shindo et al. yielded about 30/mass-ppm of copper as an impurity. Clearly, the present invention facilitates separating copper from the aqueous solution of cobalt chloride by adjusting the concentration of hydrochloric acid from 0.1 kmol/m³ to 3 kmol/m³ to obtain a high purity cobalt with reduced copper concentration of 0.101/mass-ppm or less.

I further declare that all statements made herein of my own knowledge are true, that all statements made on information and belief are believed to be true, and that these statements were

made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both (18 U.S.C. § 1001), and may jeopardize the validity of the application or any patent issuing thereon.

Masahito Uchikoshi

Masahito Uchikoshi

September 26, 1923

Date



Table 1

Results of measurements of impurities by GDMS

	Material	Shindo's method	/mass-ppm
			Our method
Al	2.110	0.373	0.022
Ca	0.074	0.026	0.004
Cr	0.671	0.118	0.016
Cu	80.000	30.000	0.101
Fe	31.400	0.070	0.084
Mg	2.150	0.003	0.002
Mn	1.050	0.002	0.002
Mo	6.290	0.050	0.026
Na	0.083	0.005	0.001
Nb	0.060	0.001	0.001
Ni	262.000	0.080	0.060
P	0.440	0.005	0.007
Pb	0.042	0.001	0.001
Si	18.800	0.010	0.012
S	0.082	0.050	0.021
Ta	2.000	1.000	1.000
Ti	2.060	0.030	0.009
Zn	20.200	0.005	0.005
Zr	0.011	0.002	0.001
W	2.600	0.040	0.050
Purity	99.9587877	99.996815	99.9998595

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